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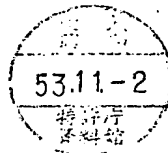
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# PATENT SPECIFICATION

(11) 1 522 686

1 522 686

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## (54) 1-(BIS-TRIFLUOROMETHYLPHENYL)-2-OXO-PYRROLIDINE-4-CARBOXYLIC ACID DERIVATIVES, THEIR PRODUCTION AND THEIR USE AS PLANT-GROWTH REGULATORS AND HERBICIDES

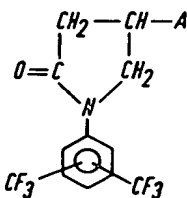
(71) We, CIBA-GEIGY AG, a body corporate organised according to the laws of Switzerland, of Basle, Switzerland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

5 The present invention relates to new 1-(bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid derivatives, to processes for their production, to compositions containing them and to their use in the regulation of plant growth and as herbicides. 5

The use of 1-phenyl-2-oxo-pyrrolidine-4-carboxylic acids, and derivatives thereof, which 10 are unsubstituted on the phenyl nucleus or substituted thereon by halogen atoms and/or a trifluoromethyl group, as active substances for the regulation of plant growth, is known from the French Patent Specification No. 1,363,615 and from the US Patent Specification No. 3,136,620. The action of these prior known compounds, such as 1- 15 (3-trifluoromethyl-4-chlorophenyl)-2-oxo-pyrrolidine-4-carboxylic acid and 1-(3,4-dichlorophenyl) 2-oxo-pyrrolidine-4-carboxylic acid, is however inadequate. Furthermore, these hitherto known compounds are in some cases phytotoxic in the usual amounts applied. 15

It has now been found that compounds of the general formula 1

20



(I)

25

20

25

wherein

A represents a group  $-COO^{\ominus}H^{\oplus}$ ,  $-COOR_1$ ,  $-COSR_2$ ,  $-CN$ ,  $-COCl$ ,

$-COF$ ,  $-CO-O-CO-R_3$ ,  $-CO-Si(CH_3)_3$ ,

$-CO-N \begin{smallmatrix} R_4 \\ \diagup \\ H_5 \end{smallmatrix}$  or  $-CO-NH-N \begin{smallmatrix} R_4 \\ \diagup \\ H_5 \end{smallmatrix}$ ,

$R_1$  represents the equivalent of a mono- to trivalent metal cation or of an inorganic or organic amine cation or quaternary ammonium cation, or an unsubstituted or substituted  $C_1$ - $C_{18}$ -alkyl radical, an alkenyl, halogenoalkenyl, alkynyl or halogeno-alkynyl radical having 3 to 10 e.g. 3 to 8, carbon atoms, a cyclo-alkyl radical having 3 to 12 ring carbon atoms, or optionally substituted phenyl, benzyl or phenethyl,  $R_2$  represents an alkyl radical having 1 to 6 carbon atoms or an optionally substituted phenyl or benzyl radical,  $R_3$  represents a  $C_1$ - $C_4$ -alkyl radical optionally substituted by at most 3 halogen atoms, or phenyl,  $R_4$  and  $R_5$  each independently represent hydrogen, optionally substituted straight-chain or branched-chain alkyl having 1-6 carbon atoms,  $C_3$ - $C_8$ -alkenyl, cycloalkyl having 3 to 8 ring carbon atoms, or optionally substituted phenyl, or  $R_4$  and  $R_5$  together with the adjacent nitrogen atom represent an optionally substituted, saturated 3- to 8-membered heterocyclic ring, which may contain a further hetero atom, have a substantially better and more flexible sphere of action than that of the prior known compounds.

Alkyl groups denoted by the various symbols  $R_1$  to  $R_3$  can be straight-chain or branched-chain.

Alkyl groups  $R_1$ ,  $R_4$  and  $R_5$  can contain as substituents, for example, 1-3 halogen atoms, especially fluorine, chlorine or bromine atoms, hydroxyl, alkoxy, alkylthio, amino, mono- or dialkyl-substituted amino groups, or a cyano, alkoxy-carbonyl, carbamoyl, tetrahydrofuryl, tetrahydro-pyranlyl or oxacyclopropyl group, with the alkyl moieties in these substituents having 1-4, particularly 1 or 2, carbon atoms.

Substituents of phenyl, benzyl or phenylethyl groups denoted by  $R_1$ ,  $R_2$ ,  $R_4$  or  $R_5$  are, e.g., alkyl, alkoxy, alkylthio, N-alkylamino or N,N-dialkylamino groups each having 1-4 carbon atoms in the alkyl or alkoxy moieties, halogen atoms, particularly chlorine or fluorine, trifluoro-methyl, amino and nitro groups.

Halogen substituents on alkenyl or alkynyl groups  $R_1$  and on alkyl groups  $R_3$  are, e.g., bromine, especially however fluorine or chlorine.

If  $R_4$  and  $R_5$  together with the nitrogen atom to which they are bound form a substituted heterocyclic ring, such as piperidine, piperazine or morpholine, then substituents are, in particular, alkyl groups having 1-4 carbon atoms.

Preferred compounds of the formula 1 are those wherein the two trifluoromethyl groups are in the 3- and 5-position of the phenyl group.

The symbol A represents preferably one of the following groups:

$-COOH$ ;

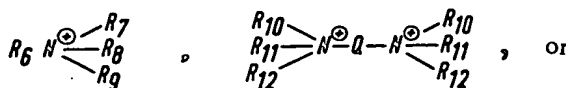
$-COOR_1$  wherein  $R_1$  represents unsubstituted alkyl having 1-6 carbon atoms, preferably 1-4 carbon atoms, especially methyl, or benzyl;

$-COSR_2$  where  $R_2$  denotes alkyl having 1-4 carbon atoms.

Further preferred compounds of the formula 1 are those wherein  $R_1$  represents an alkali metal cation, particularly the sodium or potassium cation, or the cation of an organic amine.

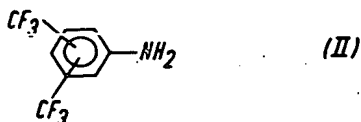
Suitable metal cations  $R_1$  are also those of alkaline-earth metals, or zinc, copper or iron. If the cation present is bi- or trivalent, then it is naturally combined with the number of anions of the parent carboxylic acid of the formula I ( $A = COO^{\ominus}$ ) corresponding to its valency. Thus  $1/n$  cation having the valency  $n$  corresponds to the parent compound I.

When A is  $-COOR_1$ ;  $R_1$  may also preferably be the equivalent of an organic amine cation or quaternary ammonium cation which is



wherein Q is C<sub>2</sub>-C<sub>10</sub> alkylene, R<sub>6</sub>, R<sub>7</sub> and R<sub>8</sub> are the same or different and are each hydrogen or C<sub>1</sub>-C<sub>12</sub> alkyl optionally substituted by up to two chlorine atoms or hydroxyl, amino, methoxy or ethoxy groups, R<sub>9</sub> is the same as R<sub>6</sub> to R<sub>8</sub> or benzyl, and R<sub>10</sub>, R<sub>11</sub> and R<sub>12</sub> are the same or different and are each hydrogen or C<sub>1</sub>-C<sub>4</sub> alkyl.

The compounds of the formula 1 according to the invention can be produced in a manner known *per se* by a process wherein an aniline of the formula II



is reacted with itaconic acid to yield a compound of the formula 1 wherein A represents the carboxyl group, and the 1-(bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid obtained is optionally converted into another derivative as defined.

The anilines of the formula II are known *per se* and can be produced in a conventional manner.

The reaction of the anilines of the formula II with itaconic acid can be performed in the melt or in an aqueous, aqueous-organic or organic medium.

Employed organic solvents can be, for example, aliphatic alcohols such as methanol and ethanol, cyclic ethers such as dioxane, aromatic hydrocarbons such as benzene and toluene, or sulpholane, pyridine and pyridine bases.

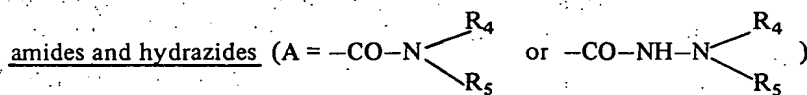
The reaction temperatures are in general between 100 and 250°C.

The conversion of the 1-(bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acids into other derivatives of the formula 1 as defined is carried out likewise in a manner known *per se*; for example

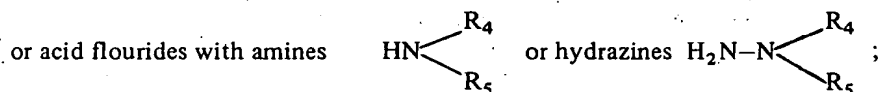
ester (A  $\eta$  COOR<sub>1</sub>)

by reaction of the free carboxylic acids with alkyl halides R<sub>1</sub>-Hal in the presence of a base; by reaction of the free carboxylic acids, or of the corresponding acid chlorides or acid fluorides, with alcohols R<sub>1</sub>OH; or by transesterification;

acid chlorides and fluorides (A  $\eta$  -COCl, -COF) by reaction of the free carboxylic acids with suitable chlorinating or fluorinating agents, such as thionyl chloride, oxalyl chloride, phosgene, PCl<sub>5</sub> or sulphur tetrafluoride (SF<sub>4</sub>);



by reaction of the free acids, acid esters, acid chlorides.



nitriles (A  $\eta$  -CN)

by dehydration of the acid amides in the presence of suitable dehydrating agents, such as P<sub>2</sub>O<sub>5</sub> or SOCl<sub>2</sub>;

Thioesters (A  $\eta$  -COSR<sub>2</sub>)

by reaction of the acid chlorides or acid fluorides with mercaptans HSR<sub>2</sub>;

mixed anhydrides (A  $\eta$  -CO-O-CO-R<sub>3</sub>)

by reaction of the free carboxylic acids, or their salts, acid chlorides or fluorides, with anhydrides (R<sub>3</sub>-CO)<sub>2</sub>;

siloxanes  $\pm \text{A} \eta$  -COO-Si(CH<sub>3</sub>)<sub>3</sub>

by reaction of the free carboxylic acids with suitable silylating agents, such as bis-(trimethylsilyl)-acetamide, trimethylchlorosilane and bis-(trimethyl)-silazane;

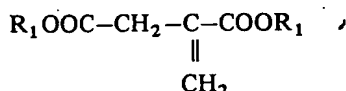
alkali metal salts, alkaline-earth metal salts, Zn-, Cu- and Fe-salts

by reaction of the free carboxylic acids with alkali metal or alkaline-earth metal hydroxides, alcoholates or carbonates, such as Na-, K-, Li-, Ca- and Mg-hydroxide, sodium and potassium methylate and sodium and potassium ethylate, and reaction of these alkali metal salts, particularly sodium salts, with inorganic, water-soluble zinc, copper and iron salts;

# ammonium salts and amine salts

by reaction of the free carboxylic acid with ammonia,  $\text{NH}_2\text{—NH}_2$ , amines having 1 or 2 nitrogen atoms which are separated by straight or cyclic alkylene bridges, or with corresponding quaternary ammonium bases of the type  $\text{N}^+(\text{R})_4\text{—OH}^-$  or  $(\text{R})_3\text{N}^+\text{—R—N}^+(\text{R})_3(\text{OH})_2^-$  in a molar ratio of 1:1 or 2:1.

Esters of the formula I ( $\text{A} = \text{—COOR}_1$ ) can be obtained also by reaction of an aniline of the formula II with an itaconic acid diester of the formula III



III

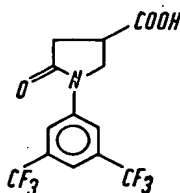
wherein  $\text{R}_1$  has the above-given meaning.

The reaction of the anilines of the formula II with the itaconic acid diesters is advantageously performed in the melt, or in an inert organic solvent, e.g. aliphatic alcohols, at a temperature of between 100 and 250°C.

After completion of the reaction, the compounds of the formula I are isolated and purified in the usual manner, e.g. by dissolving and reprecipitating, or by filtration and recrystallisation from suitable solvents such as diethyl ether, n-hexane or aliphatic alcohols having 1-4 carbon atoms.

The following Examples illustrate the production of some compounds of the invention.

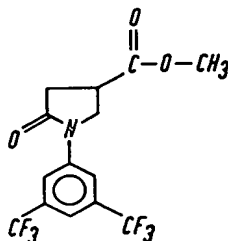
## Example 1



229 g (1 mole) of 3,5-bis-(trifluoromethyl)-aniline and 136.5 g (1.05 moles) of itaconic acid are stirred in 55 ml of water during 24 hours at 200°C in an autoclave. The reaction mixture is subsequently cooled to 20°C and 2 litres of cold water are added. Two litres of water are then evaporated, and the reaction mixture is again cooled to 20°C. The crystals that have precipitated are filtered off, dried, dissolved in 400 ml of diethyl ether and precipitated with 600 ml of n-hexane. There is obtained 242 g (71% of theory) of 1-(3,5-bis-trifluoro-methylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid; m.p. 108-9°C. Analysis for  $\text{C}_{13}\text{H}_9\text{F}_6\text{NO}_3$  (molar weight 341.22):

calculated: C 45.79% H 2.67% F 33.42% N 4.11%  
found: C 45.77% H 2.79% F 32.91% N 4.09%

## Example 2

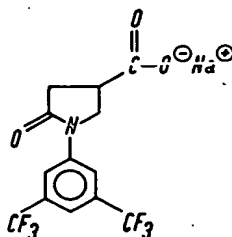


8.8 g (0.025 mole) of the 1-(3,5-bis-trifluoromethyl-phenyl) 2-oxo-pyrrolidine-4-carboxylic acid produced according to Example 1 is refluxed together with 0.3 g of p-toluenesulphonic acid in 130 ml of absolute methanol, and the reaction mixture is kept at this temperature during 17 hours. It is subsequently cooled to 20°C and 0.1 g of sodium carbonate is added. Excess methanol is evaporated off. The residue is dissolved in diethyl ether, extracted three times with water, dried, and concentrated by evaporation. By recrystallisation from n-hexane there is obtained 8.4 g (91% of theory) of

1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid methyl ester; m.p. 68-70°C. Analysis for  $C_{14}H_{11}F_6NO_3$  (molar weight 355.17):

calculated: C 47.30% H 3.12% F 32.10 3.97%  
found: C 47.42% H 3.17% F 31.97% N 4.08%

### Example 3

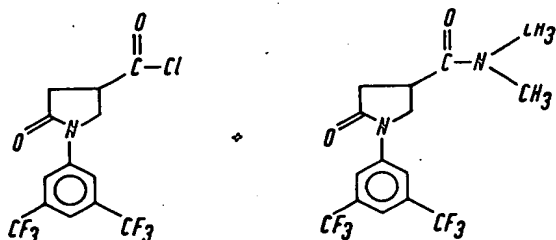


1.62 g (0.03 mole) of sodium methylate is added at 20-25°C to a solution of 10.24 g (0.03 mole) of the 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid, produced according to Exmple 1, in 50 ml of absolute methanol. The reaction is completed after 10 minutes. After removal of the methanol by evaporation, there is obtained 10.9 g (100% of theory) of the sodium salt of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid in the form of a white powder; m.p. - 310°C.

Analysis for  $C_{13}H_8F_6NNaO_3$  (molar weight 363.20).

calculated: C 42.98% H 2.22% F 31.38% N 3.86% Na 6.33%  
found: C 42.38% H 2.28% F 30.90% N 3.44% Na 6.60%

### Example 4



43 g (0.126 mole) of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid, produced according to Example 1, and 22.5 g (0.19 mole) of thionyl chloride are suspended in 135 ml of benzene, and the reaction mixture is refluxed for 18 hours. A clear solution is formed and is subsequently concentrated by evaporation. By distillation of the residue at 160°C/0.004 Torr, there is obtained 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid chloride in a 90% yield.

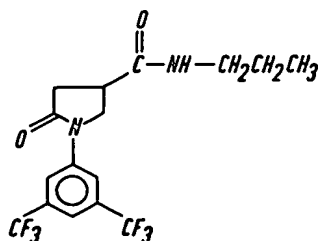
40.6 g (0.113 mole) of the acid chloride produced in this manner is dissolved in 400 ml of benzene. The reaction solution obtained is treated at 0°C with gaseous N,N-dimethylamine.

The resulting precipitate is filtered off, repeatedly washed with water, dried, and recrystallized from a mixture of diethyl ether and n-hexane (volume ratio 40:60). There is obtained 26.4 g (63.5% of theory) of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid-N,N-dimethylamide; m.p. 84.5 - 86°C.

Analysis for  $C_{15}H_{14}F_6N_2O_2$  (molar weight 368.30):

calculated: C 48.91% H 3.83% F 30.95% N 7.61%  
found: C 48.82% H 3.82% F 30.82% N 7.78%

### Example 5

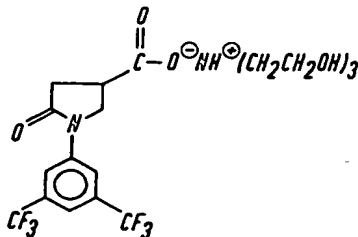


3.59 g (0.01 mole) of the 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid chloride, produced according to Example 4, paragraph 1, is dissolved in 400 ml of benzene, and 1.2 g (0.02 mole) of N-n-propylamine is added to the solution. After subsequent processing as described in Example 4 there is obtained 3.1 g (81% of theory) of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid-N-n-propylamide; m.p. 152.5 – 154°C.

Analysis for  $C_{16}H_{16}F_6N_2O_2$  (molar weight 382.32):

calculated: C 50.26% H 4.22% F 29.81% N 7.32%  
found: C 49.85% H 4.29% F 28.85% N 7.15%

#### Example 6

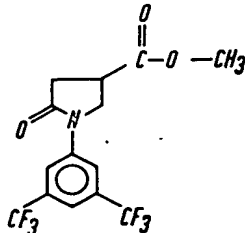


14.9 g (0.1 mole) of triethanolamine is added dropwise in the course of 15 minutes, with vigorous stirring, to a solution of 34.1 g (0.1 mole) of the 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid, produced according to Example 1, in 300 ml of diethyl ether. The solvent is subsequently evaporated off to obtain 49 g (100% of theory) of the corresponding triethanolamine salt in the form of a white crystalline residue; mp.p. 98-101°C.

Analysis for  $C_{19}H_{24}F_6N_2O_6$  (molar weight 490.41):

calculated: C 46.49% H 4.93% F 23.20% N 5.72%  
found: C 46.20% H 5.10% F 21.90% N 5.80%

#### Example 7



22.9 g (0.1 mole) of 3,5-bis-(trifluoromethyl)-aniline and 16.5 g (0.105 mole) of itaconic acid dimethyl ester are stirred for 48 hours at 200°C in an autoclave. The reaction mixture is subsequently cooled, and chromatographically separated through a silica gel column with a benzene/ethyl acetate mixture (volume ratio 85:15) as the eluant. The fractions containing 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid methyl ester are concentrated by evaporation, and recrystallised from n-hexane. There is obtained 4.3 g (12% of theory) of the above methyl ester; m.p. 68-70°C.

#### Examples 8 - 70

Also the following compounds of the formula 1 are produced by the process of the invention:

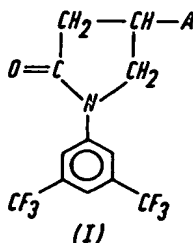



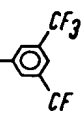
Table I: Compounds of the formula I


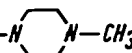

Example No.	A	m.p.( °C )
8	$-\text{COO}-(\text{CH}_2)_7-\text{CH}_3$	oil
9	$-\text{COO}-(\text{CH}_2)_{17}-\text{CH}_3$	49–50°
10	$-\text{COO}-\text{CH}_2-\text{CH}=\text{CH}_2$	oil
11	$-\text{COO}-\text{CH}_2-\text{CH}_2-\underset{\text{CH}_3}{\overset{\text{H}}{\text{C}}}-\text{CH}_2-\text{CH}_2-\text{CH}=\underset{\text{CH}_3}{\text{C}}-\text{CH}_3$	oil
12	$-\text{COO}-\text{CH}_2-\text{C}\equiv\text{CH}$	72–74°
13	$-\text{COO}-\text{CH}_2-\text{CH}_2-\text{C}\equiv\text{C}-\text{CH}_2\text{CH}_3$	64–66°
14	$-\text{COO}-\square$	oil
15	$-\text{COO}-\text{cyclododecyl}$	83–85°
16	$-\text{COO}-\text{CH}_2\text{CH}_2\text{Cl}$	oil
17	$-\text{COO}-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{Cl}$	oil
18	$-\text{COO}-\text{CH}_2\text{CH}_2\text{OCH}_3$	oil
19	$-\text{COO}-\text{CH}_2\text{CH}_2\text{OC}_4\text{H}_9$	oil
20	$-\text{COO}-\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_3$	oil



Example No.	A	m.p. (°C)
21	$\text{--COO--CH}_2\text{CH}_2\text{CH}_2\text{N} \begin{array}{l} \text{CH}_3 \\ \text{CH}_3 \end{array}$	
22	$\text{--COO--CH}_2\text{CH}_2\text{CN}$	85–86°
23	$\begin{array}{c} \text{COOC}_2\text{H}_5 \\   \\ \text{--COO--CH--CH}_3 \end{array}$	oil
24	$\text{--COO--CH}_2\text{CCl}_3$	oil
25	$\text{--COOCH}_2\text{CF}_3$	
26	$\text{--COOCH}_2\text{--CH=C--CH}_3$	oil
27	$\text{--COO--} \text{C}_6\text{H}_5$	120–122°
28	$\text{--COO--} \text{C}_6\text{H}_3(\text{Cl})_2$	125–127°
29	$\text{--COO--} \text{C}_6\text{H}_4\text{NO}_2$	79–80°
30	$\text{--COO--} \text{C}_6\text{H}_4\text{N}(\text{CH}_3)_2$	111–112°
31	$\text{--COO--CH}_2\text{--} \text{C}_6\text{H}_5$	oil

Example No.	A	m.p. (°C)
32	$-\text{COO}-\text{CH}_2-\text{C}_6\text{H}_4-\text{OCH}_3$	88-90°
33	$-\text{COO}-\text{CH}_2-\text{CH}_2-\text{C}_6\text{H}_5$	90-91°
34	$-\text{COO}-\text{CH}_2-\text{C}_4\text{H}_7$	
35	$-\text{COO}-\text{CH}_2-\text{C}_6\text{H}_{11}$	oil
35a	$-\text{COO}-\text{CH}_2-\text{CH}(\text{O})\text{CH}_2$	oil
36	$-\text{COS}-n-\text{C}_4\text{H}_9$	$n_D^{20} = 1.4930$
37	$-\text{COS}-\text{CH}_2-\text{Cl}$	58-62°
38	$-\text{COS}-\text{C}_6\text{H}_5$	91-93°
39	$-\text{CN}$	143-146°
40	$-\text{CO}-\text{O}-\text{CO}-\text{CH}_3$	
41	$-\text{CO}-\text{O}-\text{CO}-\text{CHCl}_2$	
42	$-\text{COO}-\text{Si}(\text{CH}_3)_3$	
43	$-\text{CO}-\text{NH}-\text{CH}(\text{CH}_2\text{CH}_3)_2$	135-137°

Example No.	A	m.p. (°C)
44	$\begin{array}{c} \text{CN} \\   \\ -\text{CO}-\text{NH}-\text{C}-\text{CH}_3 \\   \\ \text{CH}_3 \end{array}$	158–160°
45	$-\text{CO}-\text{NH}-\text{CH}_2\text{CH}_2\text{CH}_2\text{OCH}_3$	130–132°
46	$\begin{array}{c} \text{CH}_2\text{CH}_2\text{OH} \\   \\ -\text{CO}-\text{N} \\   \\ \text{CH}_2\text{CH}_2\text{OH} \end{array}$	88–90°
47	$\begin{array}{c} \text{CH}_3 \\   \\ -\text{CO}-\text{NH}-\text{CH}_2\text{CH}_2\text{CH}_2\text{N} \\   \\ \text{CH}_3 \end{array}$	141–142°
48	$-\text{CO}-\text{NH}-\text{CH}_2-\text{CH}=\text{CH}_2$	152–154°
49	$-\text{CO}-\text{NH}-\triangle$	165–169°
50	$\begin{array}{c} -\text{CO}-\text{N} \\   \\ \text{CH}_3 \end{array}$ 	$n_D^{20} = 1.5020$
51	$-\text{CO}-\text{NH}-\text{C}_6\text{H}_3(\text{CF}_3)(\text{CF})$ 	
52	$-\text{CO}-\text{NH}_2$	223°
53	$-\text{CO}-\text{NH}-\text{NH}_2$	280–283°
53a	$-\text{CO}-\text{NH}-\text{CH}_2-\text{CH}_2\text{OH}$	154–156°

Example No.	A	m.p. (°C)
54	$-\text{CO}-\text{NH}-\text{N}(\text{CH}_3)_2$	188°
55	$-\text{CO}-\text{NH}-\text{N}$ 	139°
56	$-\text{CO}-\text{NH}-\text{N}$ 	184°
56a	$-\text{CO}-\text{N}$ 	82°

Example 57:

1-(3,4-bis-trifluoromethylphenyl)-2-oxo-pyrrolidine-4-carboxylic acid

Table II: Compounds having cations  $\text{R}_1$ 

(n = valency)

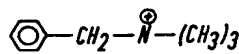
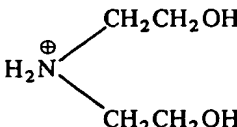
Example No.	n	Cation $\text{R}_1$	m.p. (°C)
58	2	$\text{Cu}^{\oplus\oplus}$	260-264°
59	2	$\text{Zn}^{\oplus\oplus}$	134-136°
60	1	$\text{N}^{\oplus}(\text{n}-\text{C}_4\text{H}_9)_4$	oil
61	1		resin
62	1		102°

Table II

Example No.	n	R <sub>1</sub>	m.p. (°C)
63	i	$\text{H}_3\text{N}^+-\text{n}-\text{C}_{12}\text{H}_{25}$	89°
64	1	$\text{NH}_4^+$	123-125°
65	1	$\text{CH}_3$ $\text{HN}^+-\text{CH}_3$	
66	2	$\text{CH}_2\text{CH}_2\text{Cl}$ $\text{H}_3\text{N}^+-\text{CH}_2\text{CH}_2-\text{NH}_3^+$	164°
67	2	$\text{H}_3\text{N}^+-(\text{CH}_2)_6\text{NH}_3^+$	203-205°
68	2	$\text{H}_3\text{N}^+-(\text{CH}_2)_{10}\text{NH}_3^+$	184-186°
69	2	$(\text{C}_2\text{H}_5)_2\text{HN}^+-\text{CH}_2\text{CH}_2-\text{NH}^+(\text{C}_2\text{H}_5)_2$	
70	2	$(\text{CH}_3)_2\text{HN}^+-(\text{CH}_2)_6-\text{NH}^+(\text{CH}_3)_2$	119°

The active substances of the invention influence the growth of plants in various ways. They thus inhibit, retard or prevent in the first place growth and germination. In the usual amounts applied, the compounds of the formula I are practically nonphytotoxic to the emerged plants, but they inhibit the growth in height in the case of various species of plants.

5 Compositions according to the invention which contain as active constituent at least one compound of the formula I are suitable, in particular, for the inhibition and control of plant growth on monocotyledonous and dicotyledonous plants, such as grasses, shrubs, trees, crops of cereals and leguminosae, sugar cane, tobacco, soya beans, onion and potato tubers, ornamental plants, fruit trees and grape vines.

10 By a process for inhibition of plant growth is meant a regulation of the natural development of the plant without changing the life cycle of the plant, determined by genetic properties, in the sense of a mutation. In the development of the plant (growth in height, formation of side shoots, new sprouting, blossom and fruit setting) there can be produced artificially retarding phases. The process of growth regulation is applied during the development of the plant at a point of time to be determined in the individual case.

15 The active substances of the formula I can be applied before or after the emergence of the plants; for example directly to the seeds or to young seedlings, to roots, tubers, stems, leaves or flowers or to other parts of plants. This can be effected, e.g., by application to the plants of the active substance alone or of a composition containing the active substance, and/or by treatment therewith of the nutrient medium of the plant (soil).

20 The effect primarily achieved by the compounds of the formula I is the desired reduction of the size of the plant, especially of the growth in height. There is hence in general associated with this effect a certain modification of the form of the plant. As a direct consequence of the reduction of the growth in height there occurs a strengthening of the plant. Stronger leaves and petioles are formed. As a result of a shortening of the internode distances on monocotyledonous plants, the resistance to breaking or kinking is increased.

25 As a result of these effects, crop losses due to thunder storms, and continuous rain, which normally lead to a flattening of crops of cereals and leguminosae, are largely prevented, and consequently the work of harvesting is made easier. A secondary effect is that the reduced growth in height of useful plants results in a saving of fertilisers. This applies to the same

30

extent also to ornamental plants, ornamental lawns or sports fields or to other grassland areas.

One of the most difficult problems in connection with entirely grassed areas is however, the cutting of the grass itself, whether it be grass in parks in residential districts, grass on industrial sites, on sports fields, along highways, on airfields, railway embankments or on embankments of waterways. In all these cases, a periodic cutting of the lawns or of the grass growing elsewhere is essential. This is very expensive in terms of labour and of machines, and, in addition, involves where road traffic is circulating considerable risks for the personnel concerned and for the road users. It is therefore precisely in areas with large traffic networks that there exists an urgent need on the one hand to maintain and cultivate the turf that is so necessary with regard to the consolidating of grass verges and embankments along public routes, and on the other hand to keep the grass by simple means at a moderate height during the entire vegetation period. This requirement is satisfied in a very favourable manner by application of active substances of the formula I according to the invention.

In an analogous manner, cutting work involving considerable labour costs can be reduced by the treatment of trees, shrubs and hedges, particularly in residential and industrial districts, with compounds of the formula I according to the invention.

Also the growth of shoots and/or the fruit-bearing capacity of fruit trees and grape vines can be advantageously influenced by the use of active substances of the formula I according to the invention.

Ornamental plants having a strong growth in height can be grown as compact pot plants by treatment with active substances of the invention.

The active substances of the formula I are applied also for inhibiting the growth of undesired side shoots, e.g. in the case of tobacco and ornamental plants, as a result of which the labour-intensive operation of pinching out these side shoots by hand is avoided; they are used also to inhibit the growth of side shoots on tubers in storage, for example in the case of ornamental-plant bulbs, onions and potatoes; and finally for increasing the yield of cultivated plants growing in an intensely vegetative form, such as soya beans and sugar cane, by accelerating, as a result of this application of active substances according to the invention, the transition from the vegetative phase to the generative phase, so that by reduction of the vegetative growth the generative growth is promoted.

Preferentially, the compounds of the formula I according to the invention are used for inhibition of the growth in grasses and cereal crops, as well as for the favourable regulation of growth in the case of tobacco plants, soya bean plants and cultivated plants. Some of the new active substances also exhibit a fruit-abscission effect.

The compounds of the invention may also be employed, however, for the control of weeds, in the pre-emergence or post-emergence process, in various cultivated crops, such as maize, rice, cotton, sorghum, and lucerne, particularly for the control of the weeds *Avena fatua* and *Cyperus esculentus*.

The amounts applied vary and are dependent on the point of time of application. They are in general between 0.1 and 5 kg of active substance per hectare; with application before emergence of the plants and for the treatment of existing crops they are preferably up to 4 kg per hectare.

Some of the active substances of the invention further exhibit, pronounced to a greater or lesser degree, the property of *translocation*, which is of value for the control of weeds: Applied in a test on young soya plants to the upper leaves, these active substances migrate in the sap stream of the plant (phloem) downwards and not only cause the upper parts to die off by contact action but prevent also the sprouting of new axillary buds, or effect the dying off of existing axillary buds, on the branchings of the lower sections of the stems. This property opens up interesting aspects for the control of perennial weeds, particularly if, as a result of translocation, there can be achieved a migration of active substance from the leaves down to the roots.

In accordance with the above the invention includes a method for the regulation of plant growth, which method comprises applying to the plant, to the nutrient medium of the plant or its reproductive precursor, a compound of the formula I.

The invention also includes a method for the total or selective control of weeds at a locus, which method comprises applying to the locus, before or after emergence of weeds and/or cultivated plants, a compound of the formula I.

The compositions of the invention are produced in a manner known *per se* by the intimate mixing and grinding of active substances of the formula I with suitable carriers, optionally with the addition of dispersing agents or solvents that are inert to the active substances.

The invention, therefore, includes a plant-growth regulating or herbicidal composition, which composition comprises, as active ingredient a compound of the formula I, together with a solid extender; a solid extender and a surface active agent; or a liquid diluent and a surface active agent.

The active substances can be obtained and used in the following forms:

*solid preparations:* dusts, scattering agents, granulates  
(coated granulates, impregnated  
granulates and homogenous granulates);

5 *Water-dispersible active-substance concentrates:*  
wetable powders, pastes and emulsions;

*liquid preparations:* solutions.

The solid preparations (dusts, scattering agents and granulates) are produced by the mixing of the active substances with solid carriers. Suitable carriers are, for example, kaolin, talcum, bole, loess, chalk, limestone, ground limestone, Attaclay, dolomite, diatomaceous 10 earth, precipitated silicic acid, alkaline-earth silicates, sodium and potassium aluminium silicates (feldspars and mica), calcium and magnesium sulphates, magnesium oxide, ground synthetic materials, fertilisers such as ammonium sulphate, ammonium phosphate, ammonium nitrate, urea, ground vegetable products such as bran, bark dust, sawdust, 15 ground nutshells, cellulose powder, residues of plant extractions, and active charcoal, alone or in admixture with each other.

Granulates can be produced by dissolving the active substances in an organic solvent, and applying the solution obtained to a granulated mineral, e.g. attapulgite,  $\text{SiO}_2$ , granicalcium or bentonite, and subsequently evaporating off the organic solvent.

20 Polymer granulates can be produced by impregnating, for example, a finished porous polymer granulate, such as urea/formaldehyde polymerisates, polyacrylonitrile and polyster, having a specific surface area and a favourable predetermined absorption/desorption ratio, with the active substances, e.g. in the form of their solutions (in a low-boiling) solvent, and removing the solvent. Such polymer granulates can be applied in the form of microg- 25 ranules having bulk weights of preferably 300 g/litre to 600 g/litre also by means of sprayers. Spraying can be carried out over extensive areas to be treated by the use of aeroplanes.

Granulates are obtainable also by compacting the carrier material with the active substances and additives, and subsequently crushing the compacted material.

30 It is possible to add to these mixtures also additives stabilising the active substance, and/or nonionic, anionactive and cation-active substances which improve, e.g., the adhesiveness of the active substances on plants and parts of plants (adhesives and agglutinants), and/or ensure better wettability (wetting agents) as well as dispersibility (dispersing agents). Suitable adhesives are, for example, olein/lime mixture, cellulose derivatives (methyl cellulose, 35 carboxymethyl cellulose), hydroxy-ethylene glycol ethers of monoalkyl and dialkyl phenols having 5 to 15 ethylene oxide radicals per molecule and 8 to 9 carbon atoms in the alkyl radical, lignin-sulphonic acid, the alkali metal salts and alkaline-earth metal salts thereof, polyethylene glycol ethers (Carbowaxes - registered Trade Mark), fatty alcohol polyglycol ethers having 5 to 20 ethylene oxide radicals per molecule and 8 to 18 carbon atoms in the 40 fatty alcohol moiety, condensation products of ethylene oxide, propylene oxide, polyvinylpyrrolidones, polyvinyl alcohols, condensation products of urea and formaldehyde, as well as latex products.

Water-dispersible concentrates of active substances, i.e. wettable powders, pastes and emulsion concentrates, are agents which can be diluted with water to give any desired 45 concentration. They consist of active substance, carrier, optionally additives stabilising the active substance, surface-active substances, and anti-foaming agents and, optionally, solvents.

The wettable powders nad pastes are obtained by the mixing and grinding of the active substances with dispersing agents and pulverulent carriers, in suitable devices, until 50 homogeneity is obtained. Suitable carriers are, e.g., those previously mentioned in the case of solid preparations. It is advantageous in some cases to use mixtures of different carriers. As dispersing agents it is possible to use, for example, condensation products of sulpho- nated naphthalene and sulphonated naphthalene derivatives with formaldehyde, condensa- 55 tion products of naphthalene or of naphthalenesulphonic acids with phenol and formaldehyde, as well as alkali metal salts, ammonium salts and alkaline-earth metal salts of ligninsulphonic acid, also alkylarylsulphonates, alkali metal salts and alkaline-earth metal salts of dibutyl naphthalenesulphonic acid, fatty alcohol sulphates such as salts of sulphated hexadecanols or heptadecanols, and salts of sulphated fatty alcohol polyethylene glycol ethers, the sodium salt of oleyl methyl tauride, ditertiary acetylene glycols, dialkyl dilauryl 60 ammonium chloride, and fatty acid alkali-metal and alkaline-earth metal salts.

Suitable anti-foaming agents are, e.g., silicones.

The active substances are so mixed, ground, sieved and strained with the above-mentioned additives that the solid constituent in the case of wettable powders has a particle size not exceeding 0.02 to 0.04 mm, and in the case of pastes not exceeding 0.03 mm. For 65 the preparation of emulsion concentrates and pastes, dispersing agents are used such as

those mentioned in the preceding paragraphs, organic solvents and water. Suitable solvents are, e.g., alcohols, benzene, xylenes, toluene, dimethylsulphoxide, N,N-dialkylated amides and trialkyl-amines. The solvents must be practically odourless, nonphytotoxic, inert to the active substances and not readily combustible.

5 Furthermore, the compositions according to the invention can be used in the form of solutions. For this purpose, the active substance, or several active substances, of the general formula I is, or are, dissolved in suitable organic solvents, solvent mixtures, water, or mixtures of organic solvents with water. As organic solvents, it is possible to use aliphatic and aromatic hydrocarbons, their chlorinated derivatives and alkylnaphthalenes, singly or  
10 in admixture with each other.

Other biocidal active substances or agents can be mixed with the described compositions of the invention. For the broadening of their sphere of action, the new compositions can for example contain, in addition to the stated compounds of the general formula I, insecticides, fungicides, bactericides, fungistatics, bacteriostatics, or nematocides. The compositions of  
15 the invention may also contain fertilizers, and trace elements.

The content of active substance in the above described compositions is between 0.1 and 95%, preferably between 1 and 80%. Preparations to be applied can be diluted down to 0.001%. The amounts applied are as a rule between 0.1 and 10 kg of active substance per hectare, preferably between 0.25 and 5 kg of active substance per hectare. The active  
20 substances of the formula I can be formulated, for example, as follows (parts denote parts by weight):

*Dusts:*

The following substances are used to produce a) a 5% dust and b) a 2½% dust:  
25 a) 5 parts of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid, 95 parts of talcum;  
b) 2 parts of the sodium salt of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid,  
1 part of highly dispersed silicic acid,  
97 parts of talcum.

30 The active substances are mixed and ground with the carriers.

*Granulate:*

The following substances are used to produce a 5% granulate:  
5 parts of 1-(3,5-bis-trifluoromethylphenyl)-2-oxo-pyrrolidine-4-carboxylic acid methyl  
35 ester,  
0.25 part of epichlorohydrin,  
0.25 part of cetyl polyethylene glycol ether having 8 moles of ethylene oxide,  
3.50 parts of polyethylene glycol,  
91 parts of kaolin (particle size 0.3 to 0.8 mm).

40 The active substance is mixed with epichlorohydrin and dissolved with 6 parts of acetone; the polyethylene glycol and cetyl polyethylene glycol ether are then added. The solution thus obtained is sprayed onto kaolin, and the acetone is subsequently evaporated off.

*Wettable powders:*

The following constituents are used to produce a) a 50%, b) a 25% and c) a 10% wettable powder:  
45 a) 50 parts of the sodium salt of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid,  
5 parts of sodium dibutyl-naphthalene sulphonate,  
3 parts of naphthalenesulphonic acid/phenolsulphonic acid/formaldehyde condensate 3:2:1,  
20 parts of kaolin,  
50 22 parts of Champagne chalk;  
25 parts of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid-N,N-dimethylamide,  
5 parts of the sodium salt of oleylmethyl tauride,  
2.5 parts of naphthalenesulphonic acid/formaldehyde  
55 condensate,  
0.5 parts of carboxymethylcellulose,  
5 parts of neutral potassium aluminium silicate,  
62 parts of kaolin;  
10 parts of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid- $\alpha$ -  
60 butyloxyethyl ester,  
3 parts of a mixture of the sodium salts of saturated fatty alcohol sulphates,  
5 parts of naphthalenesulphonic acid/formaldehyde condensate,  
82 parts of kaolin

65 The given active substance is absorbed onto the appropriate carriers (kaolin and chalk), and the whole is subsequently mixed together and ground. There are obtained wettable



powders having excellent wettability and suspension properties. From such wettable powders there can be obtained, by dilution with water, suspensions having any desired concentration of active substance. Such suspensions are used for the control of weeds and wild grasses in cultivated crops in the pre-germination (pre-emergence) process and for the treatment of lawns. *Paste:*

The following substances are used for producing a 45% paste:

45 parts of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid-N-n-propylamide,  
5 parts of sodium aluminium silicate,  
14 parts of cetyl polyethylene glycol ether having 8 moles of ethylene oxide,  
1 part of oleyl polyethylene glycol ether having 5 moles of ethylene oxide,  
2 parts of spindle oil,  
23 parts of water,  
10 parts of polyethylene glycol.

The active substance is intimately mixed and ground in devices suitable for the purpose. There is obtained a paste from which there can be obtained, by dilution with water, suspensions of any desired concentration. The suspensions are suitable for the treatment of lawns.

#### *Emulsion concentrate*

The following constituents are mixed together to produce a 25% emulsion concentrate:

25 parts of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid allyl ester,  
5 parts of a mixture of nonylphenolpolyoxyethylene and calcium-dodecylbenzene-sulphonate,  
35 parts of 3,5,5-trimethyl-2-cyclohexen-1-one,  
35 parts of dimethylformamide.

This concentrate can be diluted with water to give emulsions of suitable concentration.

Instead of employing the particular active substance given in each of the preceding formulation examples, it is also possible to use any of the other compounds embraced by the formula I.

The effectiveness of compounds of the formula I according to the invention has been tested as follows:

#### *I. Inhibition of the growth of grasses in a greenhouse*

The grasses

*Lolium perenne,*

*Poa pratensis,*

*Festuca ovina and*

*Dactylis glomerata*

are sown, in a greenhouse, in plastic trays containing a soil/peat/sand mixture. Three weeks after emergence, the grasses are cut back to a height of 4 cm above the soil, and two days thereafter are sprayed with aqueous preparations of the active substances listed below. Converted, the amount of active substance represents 5 kg of active substance per hectare. A tray sown with grass which had not been treated serves in each case as the control specimen. The rate of growth of the grasses is examined three weeks after application of the active substances.

Active substances employed:

1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid methyl ester,  
1-(3,5-bis-trifluoromethylphenyl)-2-oxo-pyrrolidine-4-carboxylic acid,  
sodium salt of 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid, as well as the compounds of the Examples 4, 6, 10, 19, 26, 49 and 63.

In the above test, the stated compounds of the invention produce in the case of all grasses an intense to very pronounced inhibition of growth. The grasses treated with comparable, hitherto known compounds according to US Patent Specification No. 3,136,620 exhibit no, or only slight, inhibition of growth.

#### *II. Inhibition of growth of cereals*

Spring wheat "Svenno" (*Triticum aestivum*) and Spring barley "Herta" (*Hordeum vulgare*) are sown, in a greenhouse, in plastic pots containing sterilised standard soil. The plants are sprayed one week after emergence with aqueous preparations of the active substances given in Test I. Converted, the amounts of active substance correspond to 6 kg of active substance per hectare or 2 kg of active substance per hectare. Untreated plants are used as control specimens. The rate of growth of the cereal plants is determined three weeks after application of the active substances.

Compared with the untreated control plants, the cereal plants treated with the active substances of the invention display in this test a more intense green colouration. The compounds of the invention, such as the compounds of, *inter alia*, the Examples 1, 2, 3, 4, 6, 19, 49 and 63 moreover produce the desired inhibition of the growth of the internodes. The

cereal plants treated with the prior known compounds according to the US Patent Specification No. 3,136, 620 exhibited no, or only very slight, inhibition of growth.

### III. Vegetative inhibition of growth and increase of yield of soya beans

Soya beans of the variety "Grosskern" are sown, in a greenhouse, in plastic pots containing a soil/peat/sand mixture.

Two weeks after emergence, the plants are sprayed, until dripping off commences, with aqueous preparations of the active substances given in Test 1. The concentration of active substance in the spray liquor is in the one case 500 ppm and in the other case 100 ppm. Untreated plants are used as control specimens. The rate of growth of the soya plants is determined three weeks after application of the active substances.

Compared with the control plants, the plants treated with the active substances of the invention display a more intense green colouration. Furthermore, it is shown that there has occurred a medium to extensive inhibition of growth, e.g. with the compound of the Example 1. Prior known active substances according to US Patent Specification No. 3,136,620 cause in some cases serious leaf damage and/or produce no, or only slight, inhibition of growth.

### IV. Inhibition of growth of an outdoor lawn

An established lawn in the open, consisting of 20% of *Lolium perenne*, 25% of *Poa pratensis*, 45% of *Festuca rubra* and 10% of *Agrostis tenuis*, is used as a test area. With a height of growth of 9 cm; after the first cut in the spring, plots of 3 squaremetres in area are uniformly sprayed with aqueous preparations of the active substances given in the following. The amounts applied are equivalent respectively to 5 kg and 2.5 kg of active substance per hectare (AS/hectare). Untreated plots of the same size as well as strips between the individual plots are used as control areas.

The mean height in growth of the grasses above-ground in the treated and untreated plots is measured 1, 4 and 12 weeks after application of the active substances. The results are given in the following Table.

Treatment/active substance	Mean growth in height of the grasses above-ground		
	1 week after application	4 weeks after application	12 weeks after application
1-(3, 5-bis-chloro-phenyl)-2-oxo-pyrrolidine-4-carboxylic acid			
5 kg of AS/hect.	12 cm	21 cm	58 cm
2.5 kg of AS/hect.	12 cm	24 cm	61 cm
1-(3, 5-bis-trifluoro-methylphenyl)-2-oxo-pyrrolidine-4-carboxylic acid methyl ester			
5 kg of AS/hect.	10 cm	12 cm	17 cm
2.5 kg of AS/hect.	12 cm	14 cm	26 cm
untreated	12 cm	23 cm	59 cm

### V. Inhibition of side shoots on tobacco plants

Eight weeks after sowing, tobacco plants *Nicotiana tabacum* (variety Xanthi) are transplanted to pots in a greenhouse, normally watered and treated weekly with nutrient solution. Two weeks after potting, there are chosen per treatment three plants; of these plants one remains untopped and from the two others the growth tip is removed five days before treatment.

Per plant there is then sprayed, laterally from above onto the leading shoot and the upper leaf axils, 10 ml of liquor containing active substance (concentrations: 2.6; 1.3 and 0.6%)

corresponding to 6.3 and 1.5 kg per hectare in the open). A part of the liquor consequently runs down the petioles and into the remaining lower leaf axils (contact with side-shoot buds).

- 5 After setting up of the tests in the greenhouse and watering, the tests are evaluated 4 and 14 days after application of the test liquor. 5

Contact effect and systemic effect are evaluated separately.

Contact effect: Assessment of the 6 uppermost side shoots:

- 10 9 = side shoots as in the case of untreated control plant, 10  
5 = about 50% damage on side shoots,  
1 = side shoots completely destroyed.

- 15 Systemic effect: Assessment of the uppermost side shoots. 15

Same scale of values (5 = 50% inhibition of growth without contact effect),

1 = complete inhibition of growth;

contact-effect value 1 excludes sytemic effect.

- 20 Excellent results are obtained in these tests with the active substances of the invention, 20  
such as in particular with those of, *inter alia*, the Example 1, 2, 3, 6, 10, 19, 25 and 26.

Action in breadth on monocotyledonous and dicotyledonous weeds in selected useful crops

- 25 Immediately after sowing of the test plants in seed trays, the active substances are applied 25  
as an aqueous suspension, obtained from a 25% wettable powder, at two different concentrations (4 and 2 kg/hectare) to the surface of the soil. The seed trays are then kept at 22° - 23°C with 50 to 70% relative humidity. The test results are evaluated after 28 days. The following test plants are used:

Weeds

- 30 *Avena fatua*, *Alopecurus myosuroides*, *Cyperus esculentus*, *Setaria italica*, *Echinochloa crus 30*  
*galli*, *Sesbania exaltata*, *Amarantus retroflexus*, *Chrysanthemum leuc.* *Sinapis alba*, *Ipomoea*  
*purpurea*, *Pastinaca sativa*.

Cultivate plants

maize (*Zea maize*), cotton (*Gossypium herbaccara*).

- 35 The active substances of the invention have in this test an excellent action on the given 35  
test weeds.

VII. Herbicidal action of the active substances after emergence of the plants (post-emergence applications).

- 40 The test plants are treated in the 2-4-leaf stage, about 10 days after sowing, with aqueous 40  
suspensions of the active substances, obtained from 25% wettable powders. After the treatment, the plants are maintained at 22-25°C with 50 to 70% relative humidity in a greenhouse.

The following are used as test plants:

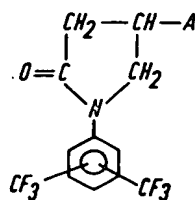
Weeds: *Avena fatua*, *Alopecurus myosuroides*, *Cyperus esculentus*, *Echinochloa crus galli*,  
45 *Sesbania exaltata*, *Sinapis alba*. 45

Cultivated plants: maize (*Zea maize*), *Sorghum hybridum*, dry rice (*Oryza*), cotton (*Gossypium herbaccara*), lucerne (*Medicago sativa*).

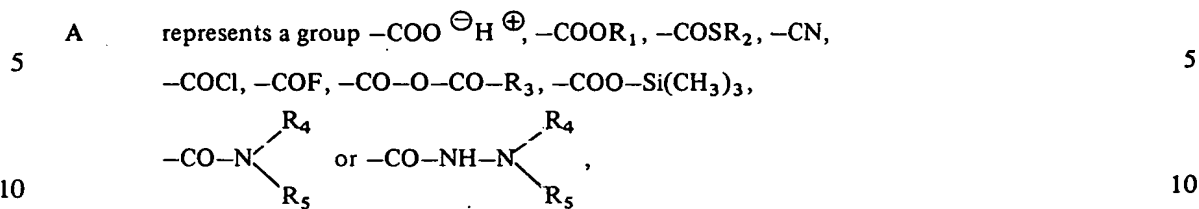
- 50 The test results are evaluated after 15 days. It is shown that the compounds of the 50  
invention, particularly 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid methyl ester, have a very good herbicidal action when applied in the usual amounts of 2 to 4 kg of active substance per hectare, with negligible damage being suffered by the cultivated plants.

WHAT WE CLAIM IS:-

1. A compound of the general formula I



wherein



$\text{R}_1$  represents the equivalent of a mono- to trivalent metal cation or of an inorganic or organic amine cation or quaternary ammonium cation, or an unsubstituted or substituted  $\text{C}_1$ - $\text{C}_{18}$ -alkyl radical, an alkenyl, halogenoalkenyl, alkynyl or halogeno-alkyl radical having 3 to 10 carbon atoms, a cyclo-alkyl radical having 3 to 12 ring carbon atoms, or optionally substituted phenyl, benzyl or phenethyl,  $\text{R}_2$  represents an alkyl radical having 1 to 6 carbon atoms or an optionally substituted phenyl or benzyl radical,  $\text{R}_3$  represents a  $\text{C}_1$ - $\text{C}_4$ -alkyl radical optionally substituted by at most 3 halogen atoms, or phenyl,  $\text{R}_4$  and  $\text{R}_5$  each independently represent hydrogen, optionally substituted straight-chain or branched-chain alkyl having 1-6 carbon atoms,  $\text{C}_3$ - $\text{C}_8$ -alkenyl, or cycloalkyl having 3 to 8 ring carbon atoms, or optionally substituted phenyl, or

$\text{R}$  and  $\text{R}'$  together with the adjacent nitrogen atom represent an optionally substituted, saturated 3- to 8-membered heterocyclic ring, which may contain a further hetero atom.

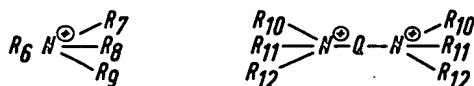
2. A compound according to claim 1 wherein the two  $\text{CF}_3$ -groups in the formula I are in the 3- and 5-positions of the phenyl radical.

3. A compound according to claim 1 or claim 2 wherein A represents the group  $\text{COOR}_1$ .

4. A compound according to claim 3 wherein  $\text{R}_1$  is an unsubstituted or substituted  $\text{C}_1$ - $\text{C}_{18}$ -alkyl radical, an alkenyl, halogenoalkyl, alkynyl or halogenoalkynyl radical having 3 to 8 carbon atoms, a cycloalkyl radical having 3 to 12 ring carbon atoms, or optionally substituted phenyl, benzyl or phenethyl.

5. A compound according to claim 3 wherein  $\text{R}_1$  is the equivalent of an alkali metal, alkaline earth metal, zinc, copper or iron cation.

6. A compound according to claim 3 wherein  $\text{R}_1$  is the equivalent of an organic amine cation or quaternary ammonium cation which is



wherein Q is  $\text{C}_2$ - $\text{C}_{10}$  alkylene,

$\text{R}_6$ ,  $\text{R}_7$  and  $\text{R}_8$  are the same or different and are each hydrogen or  $\text{C}_1$ - $\text{C}_{12}$  alkyl optionally substituted by up to two chlorine atoms or hydroxyl, amino, methoxy or ethoxy groups,  $\text{R}_9$  is the same as  $\text{R}_6$  to  $\text{R}_8$  or benzyl, and

$\text{R}_{10}$ ,  $\text{R}_{11}$  and  $\text{R}_{12}$  are the same or different and are each hydrogen or  $\text{C}_1$ - $\text{C}_4$  alkyl.

7. A compound according to claim 1 or claim 2 wherein A represents the group  $\text{COOH}$  or  $-\text{COSR}_2$ .

8. A compound according to any one of claims 1, 2 or 7, wherein A is other than  $\text{COOR}_1$  and when A includes  $\text{R}_4$  and  $\text{R}_5$  taken together with the adjacent nitrogen atom these represent an optionally substituted, saturated 3 to 8-membered heterocyclic ring.

9. The compound 1-(3,5-bis-trifluoromethylphenyl) 2-oxo-pyrrolidine-4-carboxylic acid and its sodium salt thereof.

10. A  $\text{C}_1$  to  $\text{C}_4$  alkyl ester of the compound according to claim 9.

11. The allyl ester of the compound according to claim 9.

12. The propargyl ester of the compound according to claim 9.

13. The benzyl ester of the compound according to claim 9.

14. The 2-n-butyloxyethyl ester of the compound according to claim 9.

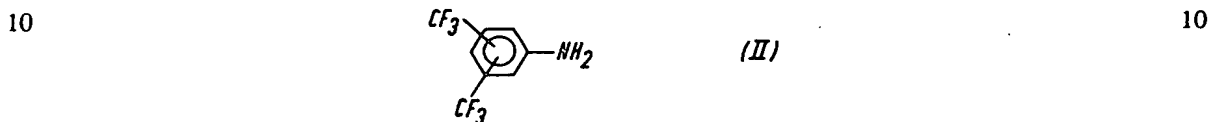
15. The tetrahydrofurfuryl ester of the compound according to claim 9.

16. A compound according to claim 1 as hereinbefore defined in any one of Examples 1 to 10, 12 to 25 and 27 to 70. 17.

17. A compound according to claim 1 as hereinbefore defined in Example 11 or Example 26.

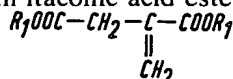
5 18. A compound according to claim 1 as hereinbefore defined in any one of Examples 35a, 53a or 56a. 5

19. A process for the production of compounds of the formula I defined in claim 1, which process comprises reacting a bis-trifluoromethyl-aniline of the formula II



15 with itaconic acid, and optionally, converting the 1-(bis-trifluoromethyl-phenyl) 2-oxo-pyrrolidine-4-carboxylic acid obtained into a salt or derivative in which A is other than -COOH. 15

20. A process for the production of compounds of the formula I defined in Claim 1 in which A is COOR<sub>1</sub>, which process comprises reacting a substituted aniline of the formula II as defined in claim 18 with an itaconic acid ester of the formula 20



25 wherein R<sub>1</sub> has, with the exception of a cation, the same meaning as that defined in claim 1. 25

21. A process according to claim 19 or claim 20 wherein 3,5-bis-trifluoromethyl-aniline is used for the reaction with itaconic acid or with an ester thereof.

30 22. A process according to claim 19 or claim 20 substantially as hereinbefore described. 30

23. A process according to claim 19 or claim 20 substantially as hereinbefore described with reference to any one of the specific Examples.

24. A compound of the formula I defined in claim 1 when produced by a process according to any one of claims 19 to 23.

35 25. A plant-growth regulating or herbicidal composition, which composition comprises, as active ingredient a compound according to any one of claims 1 to 18 or claim 24, together with a solid extender; a solid extender and a surface active agent; or a liquid diluent and a surface active agent. 35

26. A solid composition according to claim 25.

40 27. A composition according to claim 26 in the form of a granulate or pellets. 40

28. A composition according to claim 27 wherein the granules or pellets are coated or impregnated with the active ingredient.

29. A liquid composition according to claim 25.

45 30. A plant-growth regulating or herbicidal composition containing a compound according to any one of claims 1 to 18 or claim 24 and in the form of a dust, granulate, wettable powder, paste or emulsifiable concentrate specifically described herein. 45

31. A method for the regulation of plant growth, which method comprises applying to the plant, to the nutrient medium of the plant or to its reproductive precursor, a compound according to any one of claims 1 to 18 or claim 24.

50 32. A method for the total or selective control of weeds at a locus, which method comprises applying to the locus, before or after emergence of weeds and/or cultivated plants, a compound according to any one of claims 1 to 18 or claim 24. 50

33. A method according to claim 31 or claim 32 substantially as hereinbefore described.

55 Agents for the Applicants  
GALLAFENT & CO.,  
Chartered Patent Agents,  
8, Staple Inn,  
London, WC1V 7QH. 55